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JUL 76 G NORWITZ, M E EVERETT, H GORDON

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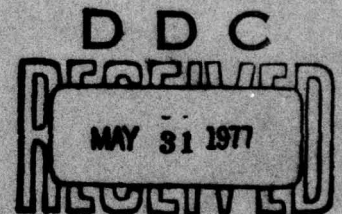
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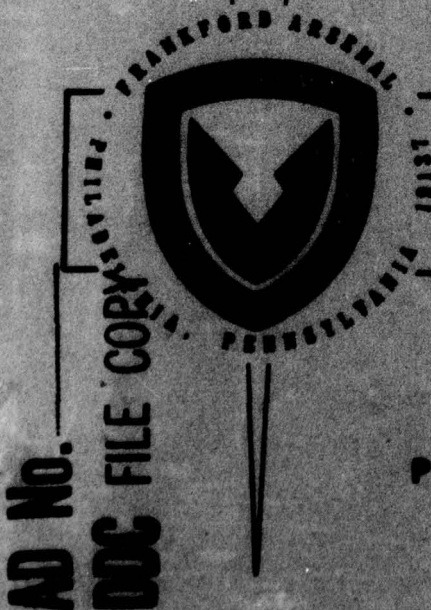
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DETERMINATION OF RUBIDIUM SALT ON COPPER-COATED
STEEL WELDING WIRE BY USE OF ATOMIC ABSORPTION

July 1976



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20. ABSTRACT: (continued)

are made at 780.0 nm using an air-acetylene flame. The potassium chloride is necessary to reduce the ionization of rubidium; without the potassium chloride, much lower absorption readings were obtained for the rubidium. It is shown the coating of rubidium carbonate produced on copper-coated steel welding wire by passing the wire horizontally through a wick saturated with rubidium carbonate solution (10%) and then through an annular compressed felt squeegee is quite variable. The results obtained ranged from a low of 0.044 to a high of 0.168 mg of rubidium carbonate per linear ft.

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INTRODUCTION

Minute amounts of rubidium salts are added to the surface of consumable wire electrodes to improve the weld by modifying the electrical and thermal properties of the arc. This laboratory was called upon to determine the amount of rubidium salt present on copper-coated steel welding process by passage through a wick saturated with rubidium carbonate solution.

Attempts were first made to determine the rubidium salt by emission spectroscopy but the results were unsatisfactory. It was found that the determination could be successfully performed using atomic absorption. Previous investigators have applied the atomic absorption technique to the determination of rubidium in such materials as water, rocks, agricultural materials, and biological materials.^{1-6*} Many investigators have observed that a large fraction of rubidium atoms are ionized in an air-acetylene flame and to eliminate this source of error they added a large excess of sodium or potassium salt.^{1-9*}

EXPERIMENTAL

Apparatus and Reagents

Perkin Elmer Model 503 atomic absorption spectrophotometer. Jarrell-Ash rubidium cathode lamp (manufactured by Westinghouse Electric Corp.), Fisher Scientific Co. JA 4-5443, Catalogue 74.

Small diameter lamp adapter kit, Perkin-Elmer 040-571.

Lamp cable (for use with octyl connectors), Perkin Elmer 303-0214.

Potassium chloride (reagent grade) in water and dilute to 1 liter.

Standard rubidium carbonate solution No.1 (1 ml = 1.0 mg Rb_2CO_3). Dissolve 1.0000 g of rubidium carbonate in water and dilute to 1 liter.

Standard rubidium carbonate solution No. 2. Pipet 20 ml of standard rubidium carbonate solution No. 1 into a 500-ml volumetric flask and dilute to the mark. Prepare fresh weekly.

Procedure

Cut off a 6-inch portion of the wire while holding the wire carefully with a cloth. Cut this 6-inch portion into about six pieces and transfer these pieces to one 50-Erlenmeyer flask.

* See References, page 6.

Add 2.0 ml of potassium chloride solution (0.75%) and 10 ml of water and boil to a volume of about 3-5 ml. Wash into a 10-ml volumetric flask with water, cool, and dilute to the mark. Prepare four standards by adding 1.0, 2.0, 3.0, and 4.0. ml of standard rubidium solution No. 2 to 10-ml volumetric flasks, adding 2.0 ml of potassium chloride solution (0.75%), and diluting to the mark. Measure the absorbance at 780.0 nm by atomic absorption using an air-acetylene flame. Determine the mg of rubidium carbonate per 10 ml and calculate the mg of rubidium carbonate per linear ft. of wire.

DISCUSSION AND RESULTS

Hollow cathode lamps and arc discharge lamps have both been used as light sources for the determination of rubidium by atomic absorption. Several years ago, it was concluded that arc discharge lamps (Osram) were advantageous for rubidium 1,4,7. However, it is the present belief that hollow cathode lamps (Westinghouse) are to be preferred.^{10, 11}

The amount of potassium chloride used in the present method to reduce the ionization of the rubidium in the flame is equivalent to 1.5 mg of potassium chloride per ml. Perkin-Elmer Corp. recommends 1-2 mg of alkali salt per ml.

Six 6-inch portions of copper-coated steel welding wire that had been coated with rubidium carbonate solution in the regular coating process by passage horizontally through a wick saturated with rubidium carbonate solution (10%) and then through an annular compressed felt squeegee were analyzed by the method. The results obtained were 0.044, 0.060, 0.168, 0.040, 0.110, and 0.112 mg of rubidium carbonate per linear ft. The results obviously showed that the coating was not uniform. The average of the results was 0.089 mg of rubidium carbonate per linear ft.

¹Perkin-Elmer Corp. "Analytical Methods for Atomic Absorption Spectroscopy", Norwalk, CN, March 1973.

⁴H. Sanui and N. Pace, Anal. Biochem., 25, 330 (1968).

⁷W. Slavin, D. J. Trent, and S. Sprague, Atomic Absorption Newsletter, 4, 180 (1965).

¹⁰Fisher Scientific Co., Catalogue 74.

¹¹Perkin-Elmer Corp., communication to Frankford Arsenal, March 1976.

The wires left in the 50-ml Erlenmeyer flask were treated with 2.0 ml of potassium chloride solution (0.75%) and 10 ml of water and the samples were again carried through the procedure. The absorbances obtained on each case were less than 0.01, so it can be deduced that all of the rubidium salt was dissolved from the wire.

Four 6-inch portions of uncoated welding wire were transferred to 50-ml Erlenmeyer flasks and 1.0, 2.0, 3.0, and 4.0 ml of standard rubidium carbonate solution No. 2 were added, followed by 2.0 ml of potassium chloride solution (0.75%) and 10 ml of water. The samples were then carried through the procedure. The recoveries were 0.046, 0.095, 0.125, and 0.196 mg, as contrasted with actual additions of 0.050, 0.10, 0.125, and 0.200 mg of rubidium carbonate. These recoveries were satisfactory.

Experiments were conducted to ascertain how much rubidium carbonate was present on wire dipped horizontally into rubidium carbonate solution (10%) and then allowed to dry. In this experiment, 8-inch portions of the copper-coated steel welding wire were pickled for about a minute in dilute hydrochloric acid (1 to 9) and then washed with water and dried with a towel. The wires were then suspended horizontally in a rubidium carbonate solution (10%) in a tray, then removed and suspended horizontally in the air for 1 hour to dry. Preliminary work had established that the coating was quite appreciable, so the rubidium carbonate was determined by treating 2-inch lengths of the wire with 5.0 ml of potassium chloride solution (0.75%) and 25 ml of water, boiling down to about 15 ml, and diluting to 25 ml in volumetric flasks. The results were 1.56, 1.98, 1.68, 1.55, and 1.61 mg of mg of rubidium carbonate per linear ft. This reproduceability was fairly good.

Some experiments were also conducted on vertical dipping, but as might be expected the variation between the top and lower portions of the wire was considerable since the solution would tend to flow towards the bottom portion of the wire.

NOTE. The lack of uniformity in the regular coating process does not indicate that this process is inadequate, since only a trace of rubidium salt need be present in the welding operation.

SUMMARY

Rubidium salt on copper-coated steel welding wire used in arc welding is determined by atomic absorption. Six-inch pieces of the wire are treated with 2 ml of potassium chloride solution (0.75%) and 10 ml of water, the solution is boiled down to 3-5 ml, the volume is brought up to 10 ml in a volumetric flask, and atomic absorption measurements are made at 780.0 nm using an air-acetylene flame. The potassium chloride is necessary to reduce the ionization of rubidium; without the potassium chloride, much lower absorption readings were obtained for the rubidium. It is shown the coating of rubidium carbonate produced on copper-coated steel welding wire by passing the wire horizontally through a wick saturated with rubidium carbonate solution (10%) and then through an annular compressed felt squeegee is quite variable. The results obtained ranged from a low of 0.044 to a high of 0.168 mg of rubidium carbonate per linear ft.

RECOMMENDATIONS

It is recommended that the method for the determination of rubidium salt on welding wire be submitted to the welding personnel for consideration as to implementation.

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